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FIELD GROUP SUB-GROUP	Tough Ceramics. Non-Destructive Evaluation. High Temperature					
A grant of \$70,000 from AFOSR was used to buy a 600W CO ₂ laser. This was combined with items bought with supplemental funds from other sources to assemble a facility for characterization of ceramic materials at extremely high temperatures (up to 2000°C is anticipated) in air. The system splits the laser beam into two equal components each of which is coupled into its own computer controlled galvanometric scanner that scans the beam rapidly over a central portion of the front or back surface of a flat specimen. The scan rate and scan density is controlled to yield a test volume, in the mid-section of the specimen, that is at uniform temperature and free of thermal stresses. Related research is developing non-contact methods for strain measurements and damage detection within the central volume of material that is at the test temperature. Characterization of material properties and behavior in a wide range of severe environments will be possible with this system.						
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AFOSR Report

Equipment Purchased:

The grant of \$70,000 for capital equipment received from AFOSR, plus the additional \$26,400 in matching funds from the Texas Engineering Extension Service (TEES), was used to purchase a large portion of the equipment for a new facility which will characterize the behavior of materials at extremely high temperatures and in severe environments.

The facility incorporates a fully instrumented, high stiffness 20,000 lb MTS hydraulic testing machine with standard water cooled grips. A straight sided flat ceramic specimen, with rectangular cross section, will be positioned in this machine for tensile loading to failure or tensile-tensile cycling. The beam from a 600 Watt CO₂ laser (Rofin Sinar Model Spectra 810) is split into two beams of equal intensity, one for the front and the other for the back surface of the specimen. Each beam is directed into a galvanometric scanner (Coneral Scanning Model LK1001) which, under computer control, scans the focused beam a raster pattern across the surface of the mid-section of the specimen. The desired scan pattern is derived from finite element predictions of the distribution of energy input needed to ensure a central test volume with uniform temperature and free of thermal stresses. Both the temperature and the uniformity of the temperature is checked with a focused beam radiometer that is manually scanned over the surface.

The AFOSR grant and the TEES cost sharing funds, supplemented with additional funds from research grants, was used to purchase the following components.

1	Rofin Sinar CO ₂ laser	74,400
1	Neslab laser heat exchanger for cooling of laser	3,490
1	Installation of heat exchanger, laser power, chilled water,	, -
	security locks and interlocks	6,490
2	General Scanning laser scanning	
	systems	31,500
	Laser safety eyewear	<u>760</u>
		\$116,640



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As a direct consequence of our ability to purchase the laser/scanner installation, we were successful in obtaining two additional grants. These were:

A gift from LTV Corporation of a large heavy duty optical table with isolation mounts, safety hood, CO₂ optical components and optical mounts with a current catalog value in excess of \$30,000.

A competitive research grant from the U.S. Department of Energy/Texas Energy Research Applications Program was awarded to us for the development of tough

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ceramics for improved fuel efficiency in thermal systems (\$206,000). This grant has been providing for additional equipment, operating expenses, and support for 4 graduate research assistants (all U.S. citizens) over the past 20 months.

Capabilities of High Temperature Facility:

Although the design specifications for this high temperature material testing facility was dictated by our estimates of the needs for characterizing the mechanical properties and to observe damage development under high temperature oxidizing conditions of the emerging generation of tough ceramics, it can do much more:

- * Since the need for a kiln to heat the specimen is eliminated, the only thermal inertia that needs to be overcome when attempting to impose rapid thermal cycling and thermal shock on a specimen, is that of the specimen itself. This provides unique opportunities for fundamental studies of the effects of such transient loads on the high temperature behavior of structural materials.
- * Since a specimen can be heated without containment, free optical access to the specimen is available. This permits the use of optical measurement methods and generally facilitates the use of non-contact approaches for studying the time temperature behavior of materials in real time, i.e. on line, while a test is in progress. This feature is particularly important when working with expensive material systems or with experimental material systems where large numbers of specimen may not be available. On-line data provide information on time dependent damage development more directly than competing test systems where specimens are fractured and the mechanics leading to failure are inferred from "after the event" observations. It is fundamentally a better approach for studying the micromechanics of the interactive effects of time, temperature, load and environment in damage initiation and growth. To achieve this capability new optical methods are needed. These are being developed in a parallel program that is closely correlated with developments in the high temperature facility.
- * Since the temperature of the environment around the specimen is relatively cool, enclosures to house adverse environments can be easily constructed while retaining optical access to the specimen. This permits elevated temperature testing in esoteric environments of reactive materials, e.g. some nuclear fuel and fuel containment materials and other material systems in severely corrosive or poison environments.

Our goal is to reach a temperature of 2000°C in the test volume. However, since the thermal properties at operating temperatures of the new materials are not known in advance, calculations of the maximum temperatures attainable with a 600W laser and without damaging the surfaces being scanned, are of varying accuracy. We are confident that 1500°C can be maintained in a reasonably sized (100x10x2 mm) specimen of ceramic/ceramic composite. Our present goal is to achieve a test volume of 20x10x2 mm.

Research in Progress:

The research on establishing the interrelationships between composition, microstructure, mechanical properties and damage development of ceramic composites in their operating environment, for which this laser based heating system was purchased, is well under way. It has four components:

- 1. Research toward establishing, through laser heating, a suitable technology for creating, within a specimen, a test volume of material that is free of thermal stresses and at a uniformly high temperature.
- 2. Research towards developing a non-contact optical "strain gauge" that can monitor strains in the test volume when the specimen is very hot and loaded mechanically and/or cycled thermally.
- 3. Research toward establishing a non-destructive, non-contact ultrasonic technique for observing and monitoring damage development in real time during thermal/mechanical testing.
- 4. Research that exploits these three new techniques toward building a fundamental understanding of the time dependent mechanisms that control the structural performance, strength and life of materials under adverse conditions. The intent is to acquire the knowledge needed to: a) design new materials that are better able to withstand adverse environments, and b) characterize the mechanical and thermal properties at operating conditions with sufficient accuracy to permit reliable design and life predictions for structures made from these materials.

The funds from AFOSR were applied to the first component in this quartet. However, all of the first three components are being addressed along three parallel tracks that will, by the end of 1990 converge into a multi-faceted approach to the last, and most important component. At present, each of the three "techniques" are progressing independent of each other. Progress is as follows:

Laser heating of test specimen (1):

The need here is for a small, but known volume of material that occupies only a portion of the specimen but extends throughout it's cross section and that is at a uniform temperature and free of thermal stresses. When the specimen is then loaded externally, the behavior of the material in the test volume will respond to the loads and the environment such that valid conclusions can be drawn from the results of measurements made on and within the volume.

Doing this is no simple matter. At play is the absorption coefficient of the surface for the wavelength of CO_2 radiation (10.6 μ m), the thermal diffusivity, the radiation coefficient of the material and the maximum permissible input energy density so that the surface is not damaged. These properties are all temperature dependent, often non-linearly, and with sudden changes at certain temperature when phase

transformations or local resonances occur. They are not even known for new materials and are seldom available for existing material systems at the temperatures anticipated in this program. For this reason a good interactive numerical model that can be tuned to correctly predict observed temperatures, temperature gradients and energy input rates is essential. Once a particular material system has thus been "matched" the model can be used to program scan rates, scan patterns and energy input rates for different desired experimental conditions on the same material. This modeling work is well under way. At present, we are calibrating everything on samples of steel and aluminum with known properties at moderate temperatures of around 500°C.

Figure 1 is a diagrammatic of the overall heating system and figure 2 is an example of an early modeling for energy input to obtain near uniform temperatures. The laser system itself together with the scanners and associated optics have been debugged (it is a shame that this should be necessary but such is the state of the technology) and the computer scan system is working. Calibration tests for the system is being performed on different materials with known properties. We have heated freely supported specimens on a table but have not yet coupled the laser beam to a specimen in the test machine.

Optical Strain Measurement at High Temperatures (2):

For this purpose we are developing a procedure that uses spatial frequency domain analysis of surface features - natural or imposed - to extract local strains. The procedure is to record, through a high quality telemicroscope lens (Questar System) into a digital video frame grabber an enlarged image of the features on the surface of the test volume. Line scans across short incremental pieces of this image provides short clips of intensity variations with distance. These plots resemble moving time domain electrical pulse bursts. They are transformed into the frequency domain in the same way as in electrical signal analysis and dominant frequencies are extracted. Thus a "frequency" map can be generated for the unloaded surface of the test volume. The specimen is then strained and the process repeated to yield a frequency map of the deformed surface. Correlation between the two images and between the two frequency maps can then be performed to sub-pixel accuracy after which the local changes in frequency can be extracted. We have performed computer simulation of this procedure in which we were able to obtain in-plane strain resolution of $10x10^{-6}$ (10 micro strains). The experimental set-up to match this performance on a specimen in a testing machine at high temperature is also not easy but we are making progress. Out best current achievement has been reliable measurements of 500 micro-strains. However, new optical elements are on order which, we believe, will allow us to achieve at least a one order of magnitude improvement.

Non-Contact NDE (3,4):

Here we are using a short pulse from a Nd-YAG laser to generate, by means of a thermal shock, an ultrasonic wave in the material. The laser pulse is guided through high energy fiber-optics so that it is available in difficult to reach locations. The

shape of this wave as well as its velocity and/or dispersion is recorded with an optical fiber based interferometer system for which we have been awarded a patent. The potential spatial resolution of this system is less than 200 µm, with a possibility to achieve 10 μ m when gradient lenses are used to condition the light spot from the interferometer. The simple system, as described here, works but the signal to noise ratios are low because the input energy disperses evenly in all directions so that the wave amplitude at the detection point is small. Current research on the generation side of this system is concentrating on spatial and temporal conditioning of the thermal pulse to be able to guide the energy in preferred directions. To this end, we are developing an interactive computer model of the coupled thermal stresses and the resulting waves so that we can design optimum shapes for the distribution of an array of fiber tips, and of the energy distributions within the array for directing the bulk of the energy in desired directions. The goal is to generate larger wave amplitudes at the points where the waves are being "read" by the interferometer. On the interferometer end of the system we are concentrating on adapting two frequency and dual fiber systems that will relax the stability and location restrictions of the current set-up. We are making good progress in both these areas.

Acknowledgements:

The support of AFOSR for the purchase of the CO₂ laser provided the base around which the research on high temperature characterization could be built.

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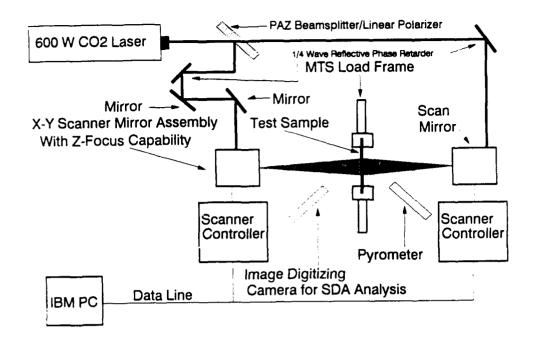


Figure 1: Schematic of system for laser heating of ceramic specimens in an MTS testing machine

